

SYMPOSIUM ON CHARACTERIZATION AND CHEMISTRY OF OIL SHALES
PRESENTED BEFORE THE DIVISIONS OF FUEL CHEMISTRY AND
PETROLEUM CHEMISTRY, INC.
AMERICAN CHEMICAL SOCIETY
ST. LOUIS MEETING, APRIL 8 - 13, 1984

THE DETERMINATION OF SULFUR SPECIES IN
OIL SHALE RETORT GAS

By

J. R. Wallace
Denver Research Institute, University of Denver
Denver, Colorado
and
E. Sexton
Chemistry Department, Pennsylvania State University
University Park, Pennsylvania

ABSTRACT

Gas chromatography with flame photometric detection was developed as a means of measuring sulfur species in retort gas. Two potential problems were considered in detail: 1) quenching of the fluorescent signal by coeluting hydrocarbons, and 2) adequate separation of the large number of sulfur species which could potentially occur in retort gas. Fluorescent quenching effects were measured on two types of commercially available FPD's, a single-flame detector and a dual-flame detector. The latter exhibited no significant quenching effects over the concentration ranges of interest in retort gas. However, for the single-flame detector quenching effects cannot be ruled out entirely. Although hydrogen sulfide in retort gas is usually abundant enough to minimize quenching effect, the minor species (COS, CH₃SH, CS₂, and CH₃CH₂SH) could be subject to quenching effects unless precautions are taken. These precautions include operating the detector with the air and hydrogen flows reversed and measuring peak height rather than peak area. The single-flame detector exhibited both suppression and enhancement of the fluorescent signal. Columns were evaluated with respect to their ability to separate the sulfur species of primary interest--H₂S, COS, CH₃SH, SO₂, CS₂, and CH₃CH₂SH--from each other as well as from the later eluting sulfur compounds and the potentially interfering hydrocarbons. Columns were also tested with respect to their ability to tolerate water vapor and for their stability during tests with actual retort gas. A method is described for the primary sulfur species in the range 5-50,000 ppmv using a Carbowack B HT 100 column arranged in a backflush-to-detector configuration.